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# Mode-Locked Er-Doped Fiber Laser by Using MoS<sub>2</sub>/SiO<sub>2</sub> Saturable Absorber

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## Abstract

The two-dimensional (2D) layered material MoS<sub>2</sub> has attracted numerous attentions for electronics and optoelectronics applications. In this work, a novel type of MoS<sub>2</sub>-doped sol-gel glass composite material is prepared. The nonlinear optical properties of prepared MoS<sub>2</sub>/SiO<sub>2</sub> composite material are measured with modulation depth ( $\Delta T$ ) of 3.5% and saturable intensity ( $I_{\text{sat}}$ ) of 20.15 MW/cm<sup>2</sup>. The optical damage threshold is 3.46 J/cm<sup>2</sup>. Using the MoS<sub>2</sub>/SiO<sub>2</sub> composite material as saturable absorber (SA), a passive mode-locked Er-doped fiber (EDF) laser is realized. Stable conventional soliton mode-locking pulses are successfully generated with a pulse width of 780 fs at the pump power of 90 mW. In the pump power range of 100–600 mW, another stable mode-locking operation is obtained. The pulse width is 1.21 ps and the maximum output power is 5.11 mW. The results indicate that MoS<sub>2</sub>/SiO<sub>2</sub> composite materials could offer a new way for optical applications.

**Keywords:** Transition metal dichalcogenides, Nonlinear optical materials, Saturable absorbers

## Introduction

Nonlinear optical materials, especially those with 2D structures, lay the foundations of optoelectronics development [1–5]. The graphene has been intensively investigated as an optical modulator for use in diverse pulsed lasers and excellent results are obtained [6, 7]. Recently, numerous novel 2D materials such as topological insulators [8, 9], transition metal dichalcogenide (TMD) [10–14], black phosphorus [15], MXene [16], bismuthene [17], metal–organic frameworks [18], and perovskite [19] have demonstrated broadband optical nonlinearities. In addition, these 2D materials are considered as the next generation promising optical modulator materials [20, 21]. The MoS<sub>2</sub> is a representative TMD semiconductor with crystal layers consisting of three alternating hexagonal planes of Mo and S [22]. Depending on the coordination and oxidation states of transition metal atoms, MoS<sub>2</sub> can either be semiconducting or metallic in nature. The broadband saturable absorption and high third-order nonlinear susceptibility have been thoroughly studied [23–25]. Recent works demonstrate that the MoS<sub>2</sub> has better saturable absorption response than graphene by using an open-aperture Z-scan technique for ultrafast

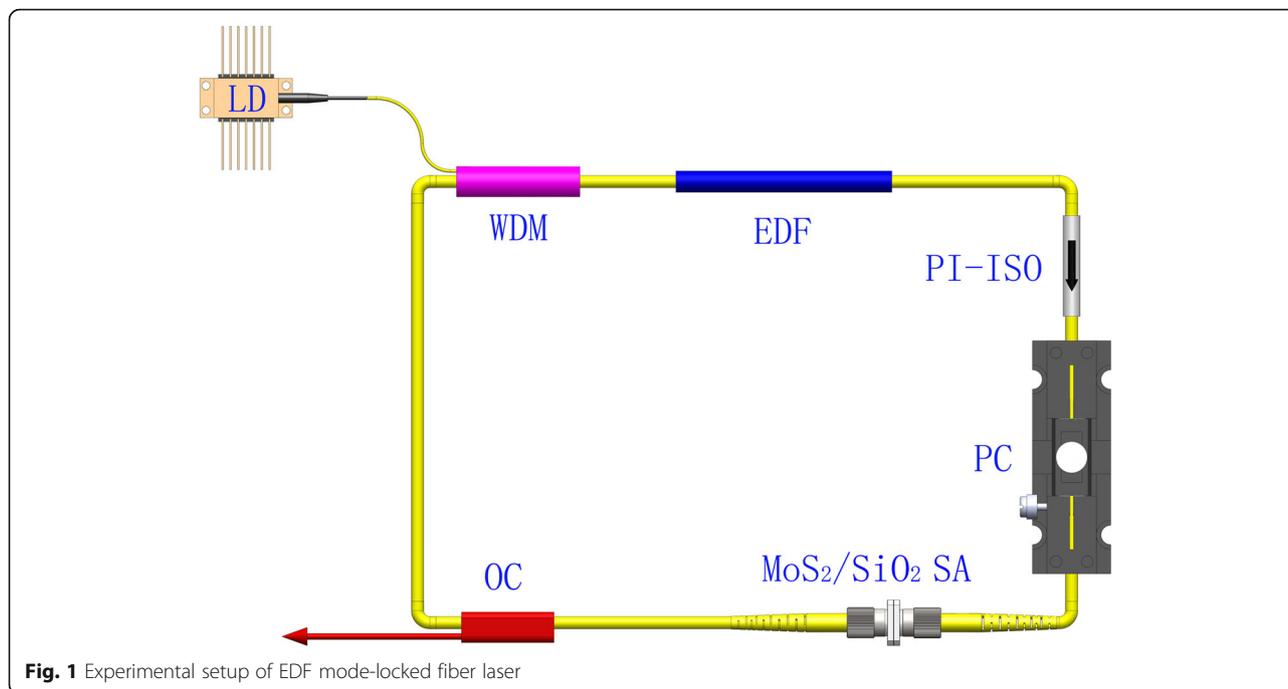
nonlinear optical properties [26, 27]. Based on the MoS<sub>2</sub> materials, the corresponding optical modulator devices have been used for pulsed lasers successfully. So far, pulsed fiber lasers with MoS<sub>2</sub> at different central wavelengths of 635 nm, 980 nm, 1030 nm, 1560 nm, 1925 nm, and 2950 nm have been achieved [28–33]. Ultrafast fiber lasers based on MoS<sub>2</sub> emitting pulses with pulse duration from hundreds of femtoseconds to few picoseconds also have been reported [34, 35]. Moreover, high repetition rate pulsed fiber lasers with MoS<sub>2</sub> have been realized [36, 37].

Usually, MoS<sub>2</sub> nanomaterials are fabricated via mechanical exfoliation (ME) method [38], liquid phase exfoliation (LPE) method [39], hydrothermal method [40, 41], chemical vapor deposition (CVD) method [42], pulsed laser deposition (PLD) method [43], and magnetron sputtering deposition (MSD) method [44]. Every method has its strengths and weaknesses. For example, ME method is the first reported technique for obtaining layered structure MoS<sub>2</sub>. However, this method has the disadvantages of poor scalability and low yield, hindering the large-scale applications. To overcome the defects of ME method, CVD offers a controllable approach for the production of single and few-layer MoS<sub>2</sub>. While for the MoS<sub>2</sub> growth, it is often necessary to pretreat of the substrate. PLD and MSD should be the ideal methods for growing high-quality MoS<sub>2</sub> film directly with different sizes and areas, but with

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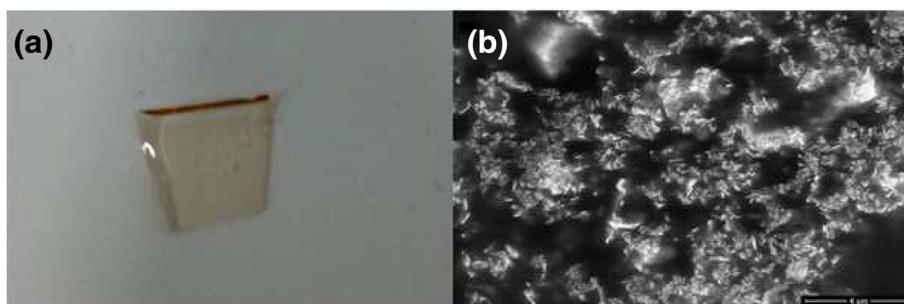
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many crystal defects. The reported technology for incorporating MoS<sub>2</sub> into fiber lasers can be mainly divided into two methods: (1) directly sandwiching the MoS<sub>2</sub>-based SAs between two fiber connectors by mixing the MoS<sub>2</sub> nanomaterials into polymer film and (2) depositing the MoS<sub>2</sub> nanomaterials on tapered fiber or D-shaped fiber by using the evanescent wave interaction. The sandwich-type MoS<sub>2</sub> optical modulators have the advantages of flexibility and convenience. It also has the weak point of low thermal damage. The evanescent wave method can enhance the damage threshold of SAs, but it has the shortcoming of frangibility. For practical applications, tapered fiber or D-shaped fiber-based optical modulators need to be packaged, which makes the fabrication procedure very complicated. Therefore, establishing fine-controlled MoS<sub>2</sub> nanomaterial still require deeper exploring, and improving effective fabrication method is still a long-standing goal.

In this paper, we demonstrate a novel method to prepare the MoS<sub>2</sub>/SiO<sub>2</sub> composite materials by doping the MoS<sub>2</sub> nanomaterials in sol-gel glass. As is well known, the sol-gel method is a mature approach to prepare the glass at low temperature [45, 46]. Doping the MoS<sub>2</sub> nanomaterials in the sol-gel glass not only has virtues of good antioxidant capacity, but also can effectively increase the mechanical stability. In addition, the sol-gel glass has a good refractive index matching with the optical fiber. Therefore, this type of composite material shows a high environmental damage threshold. By incorporating the proposed MoS<sub>2</sub>/SiO<sub>2</sub> into EDF laser cavity, we achieve two kinds of mode-locking operation. At the pump power of 90 mW, the conventional soliton mode-locking operation is obtained. The pulse duration is 780 fs. In the pump power range of 100–600 mW, we also realize another stable mode-locking operation. The pulse width is 1.21 ps and the maximum output power is 5.11 mW. The



**Fig. 2** a Digital photos. b SEM image

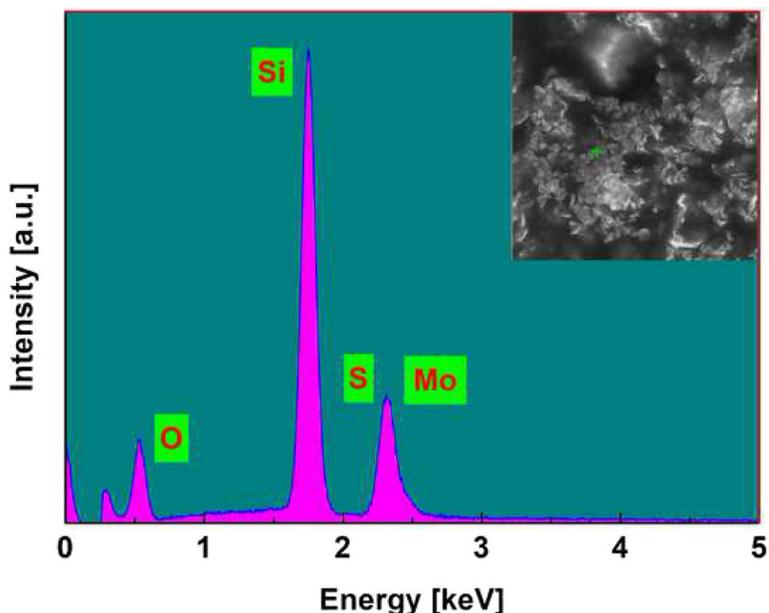


Fig. 3 EDS spectrum

results show that the MoS<sub>2</sub>/SiO<sub>2</sub> composite materials possess great potential for mode-locked fiber laser applications.

**Methods**

**MoS<sub>2</sub>/SiO<sub>2</sub> Composite Materials Preparation Procedure**

The MoS<sub>2</sub>/SiO<sub>2</sub> composite materials are prepared by the sol-gel method. In the first step, the MoS<sub>2</sub> dispersion is prepared by liquid-phase exfoliation method. One

milligram of MoS<sub>2</sub> nanosheets is put into the 10 ml deionized water. Then, the MoS<sub>2</sub> dispersion is ultrasonically for 6 h and the power of ultrasonic cleaner is set as 90 W. After the centrifugation process, we obtain the stable MoS<sub>2</sub> solution. On the other hand, the tetraethoxysilane (TEOS), ethanol, and deionized water are mixed for the sol-gel glass preparation. In the next step, the MoS<sub>2</sub> solution and the TEOS mixture are mixed. Then, the MoS<sub>2</sub> and TEOS mixture is stirred to form the

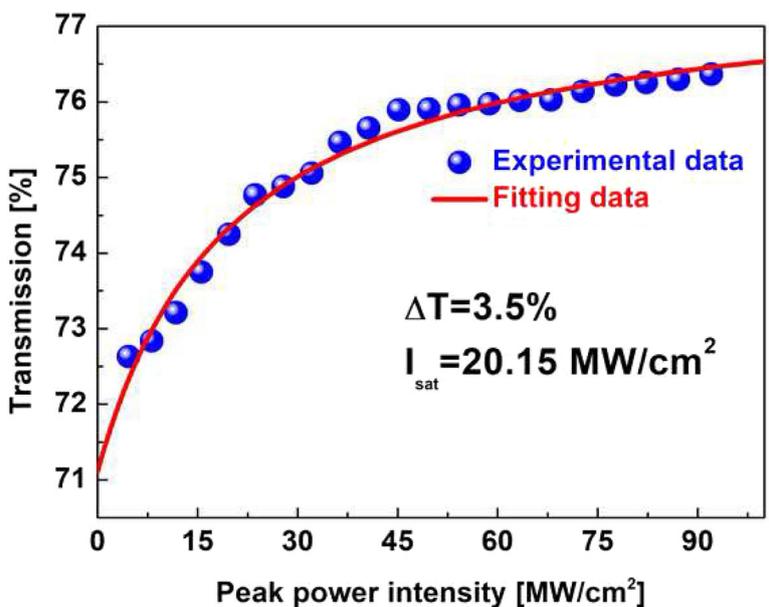
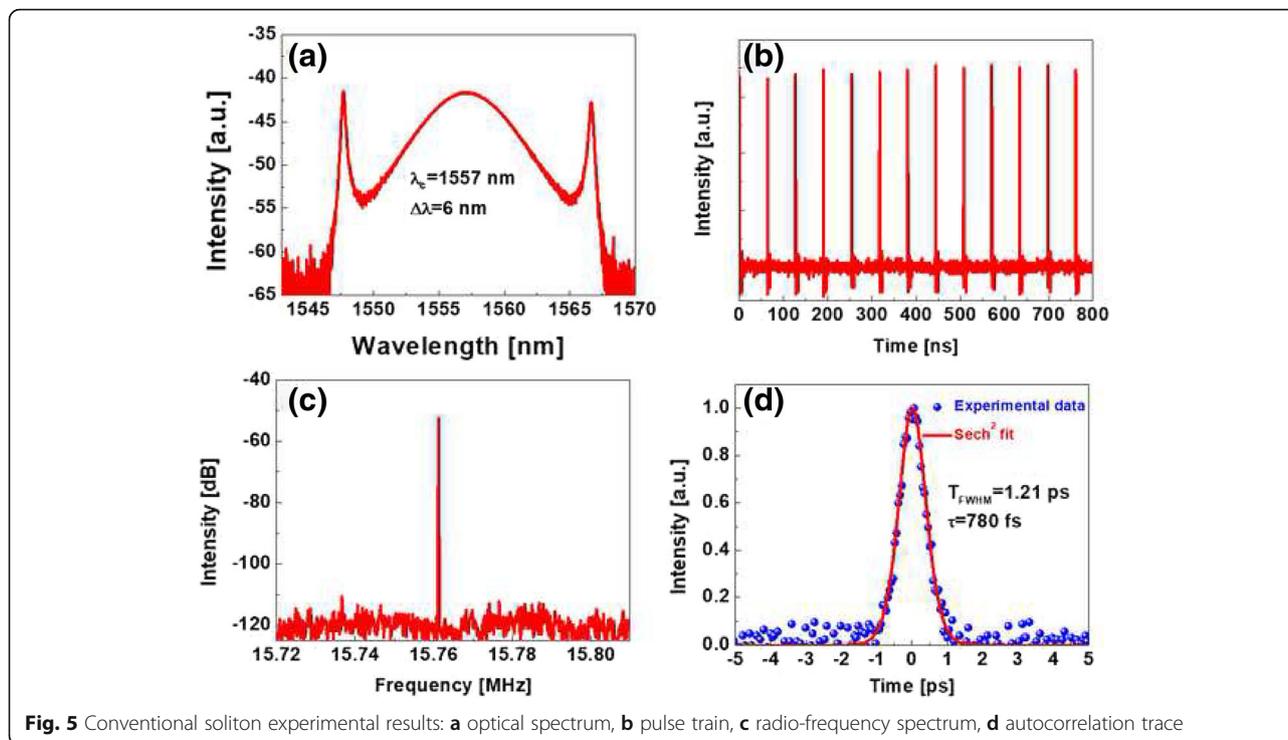
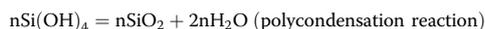
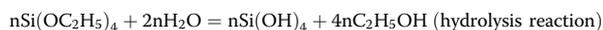


Fig. 4 Nonlinear optical properties of MoS<sub>2</sub>/SiO<sub>2</sub> composite materials



**Fig. 5** Conventional soliton experimental results: **a** optical spectrum, **b** pulse train, **c** radio-frequency spectrum, **d** autocorrelation trace

MoS<sub>2</sub>-doped glass. At this time, the hydrochloric acid is added into the obtained mixture to control the PH at low value. Via hydrolysis and polycondensation process, the MoS<sub>2</sub>-doped silica sol is obtained. The hydrolysis and polycondensation process can be described as the following reactions:



During the hydrolysis process, the alkoxide groups of the TEOS are replaced by the hydroxyl groups. In the polycondensation process, the Si-OH groups produce the Si-O-Si networks. In order to avoid the sol-gel glass cracking and MoS<sub>2</sub> agglomeration, the MoS<sub>2</sub>-doped silica sol are stirred at 50 °C for 5 h. Then, the MoS<sub>2</sub>-doped silica sol are put into the plastic cells and aged at room temperature for 48 h. In the final step, put the silica sol into a dry box at 60 °C for 1 week to form solid MoS<sub>2</sub>-doped glass.

#### Fiber Laser Cavity

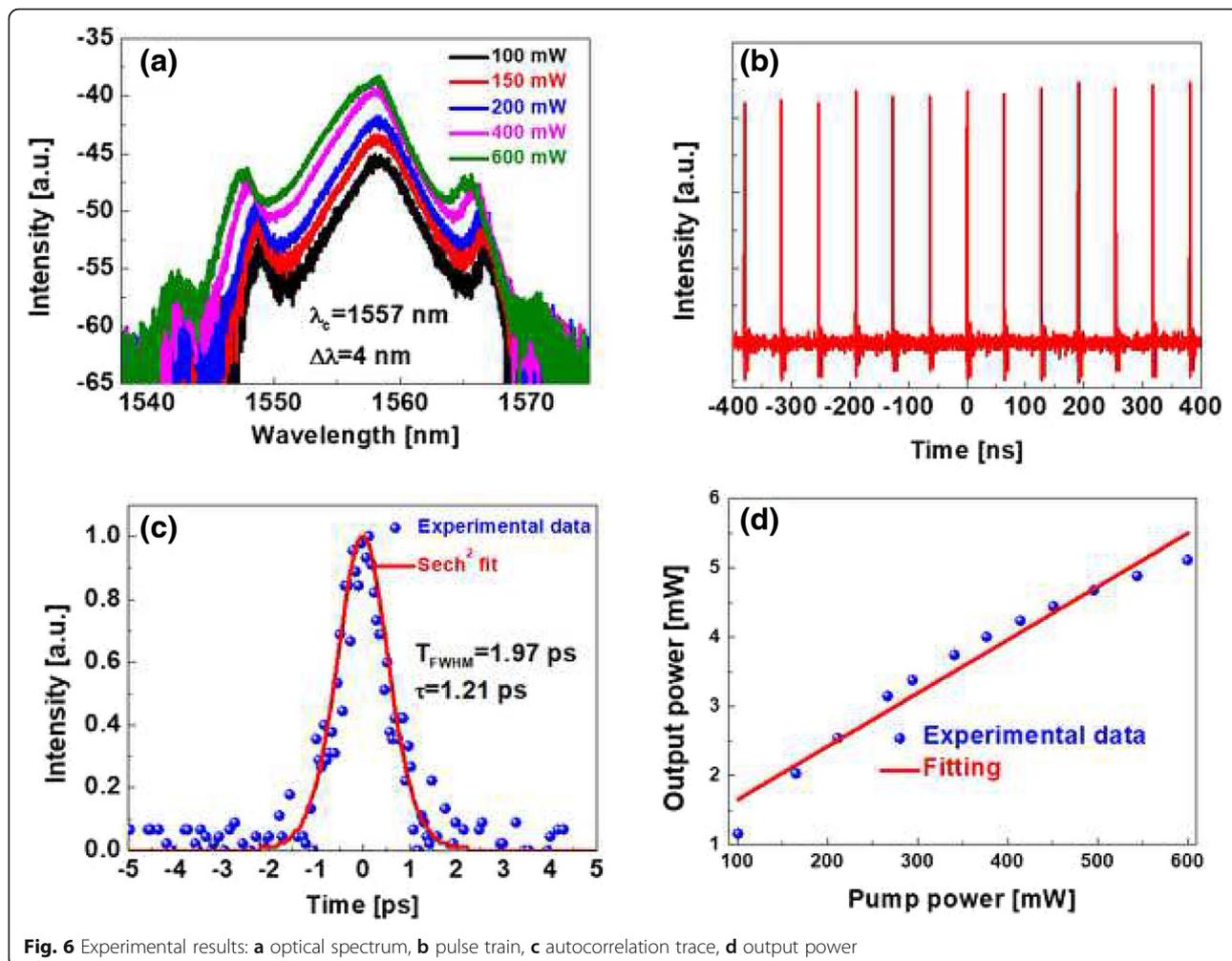
The layout of the EDF laser with MoS<sub>2</sub>/SiO<sub>2</sub> composite material is displayed in Fig. 1. The ring laser cavity is used. The pump source is a fiber-coupled laser diode (LD) with the maximum output power of 650 mW, which delivers the pump laser into the laser cavity via the wavelength division multiplexer (WDM). A 1.2-m-long EDF is employed as the gain medium. A

polarization independent isolator (PI-ISO) is used to ensure the unidirectional operation in the ring laser cavity. A polarization controller (PC) is engaged to achieve different polarization states. A MoS<sub>2</sub>/SiO<sub>2</sub> composite material is sandwiched between two fiber ferrules. The 10/90 optical coupler is used at the laser cavity output port. The total length of the laser oscillator cavity is about 13.3 m.

## Results and Discussion

### Characterization of MoS<sub>2</sub>/SiO<sub>2</sub> Composite Materials

As is shown in Fig. 2a, the prepared MoS<sub>2</sub>/SiO<sub>2</sub> composite material is the brown color, indicating the MoS<sub>2</sub> nanosheets are incorporated into the silica glass. Figure 2b shows the SEM image. The MoS<sub>2</sub>/SiO<sub>2</sub> composite material is also characterized by energy dispersive X-ray spectrometer (EDS). Figure 3 shows the EDS spectrum, which indicates that the prepared MoS<sub>2</sub>/SiO<sub>2</sub> glass contains three elements (Mo, S, and Si). The nonlinear optical properties of MoS<sub>2</sub>/SiO<sub>2</sub> glass are investigated by the balanced twin-detector measurement system. The pulse laser source is the home-made EDF fiber laser with a central wavelength of 1550 nm, pulse width of 500 fs, and repetition rate of 23 MHz. As can be seen from Fig. 4, the modulation depth ( $\Delta T$ ) and saturable intensity ( $I_{\text{sat}}$ ) are measured to be 3.5% and 20.15 MW/cm<sup>2</sup>, respectively. A femtosecond Ti:sapphire laser (central wavelength 800 nm, pulse width 250 fs, repetition rate 100 kHz) is used as the source to investigate the thermal



**Fig. 6** Experimental results: **a** optical spectrum, **b** pulse train, **c** autocorrelation trace, **d** output power

damage of MoS<sub>2</sub>/SiO<sub>2</sub> composite material. The optical damage of the MoS<sub>2</sub>/SiO<sub>2</sub> appears when the test power is adjusted to 3.46 J/cm<sup>2</sup>, which is much higher than that of semiconductor saturable absorber mirror (SESAM) (500 μJ/cm<sup>2</sup>).

#### MoS<sub>2</sub>/SiO<sub>2</sub> Mode-Locking Fiber Laser

The conventional soliton mode-locking experimental results are shown in Fig. 5. The mode-locking operation is observed at the pump power of 90 mW accompanying hysteresis phenomenon [47]. By adjusting the pump power lower to 75 mW, the mode-locking state is still maintained. The optical spectrum of mode-locking pulses at the pump power of 90 mW is depicted in Fig. 5a. The central wavelength is located at 1557 nm and the 3-dB spectral width is 6 nm. It can be seen clearly that the Kelly sidebands appeared at both sides of spectrum symmetrically, indicating the fiber laser works in conventional soliton mode-locking state. Figure 5b shows the performance of the pulse train, which has uniform intensity. The interval of two pulses is 64.2 ns,

corresponding to the cavity roundtrip time. To further study the stability of soliton pulse, the radio-frequency spectrum is measured. Figure 5c shows that the fundamental repetition rate is 15.76 MHz and the signal-to-noise ratio (SNR) is 65 dB. The pulse duration is measured by an autocorrelator. Figure 5d shows the autocorrelation curve. The full width at half maximum (FWHM) is measured to be 1.21 ps, indicating the pulse duration is 780 fs if a Sech<sup>2</sup> fit is used. We just increase the pump power to 100 mW and keep the PC unchanged, the laser enters into multiple pulses operation mode-locking regime, presenting instability and fluctuations, which means the mode-locking operates in narrow pump range.

During the experiments, we achieve another mode-locking state. By adjusting the pump power to 100 mW and the PC rotation, we obtain this mode-locking operation state. Figure 6a records the corresponding optical spectrum. The optical spectrum is getting wider and wider with pump power increasing. Gradually increasing the pump power to 600 mW, this mode-locking operation can

always be maintained. It is observed that the sides appeared in the optical spectrum with relative small intensity. The central wavelength is 1557 nm and 3-dB spectral width is 4 nm at the pump power of 600 mW. The oscilloscope trace for the mode-locking state is depicted in Fig. 6b; the interval of two pulses is 64.2 ns, verifying that the fiber laser is working in the fundamental mode-locking state. The autocorrelation trace is displayed in Fig. 6(c), the full width at half maximum (FWHM) is 1.97 ps, which means the pulse duration is 1.21 ps if a  $\text{Sech}^2$  fit is used. The average output power characteristics are shown in Fig. 6d. As the pump power increases, the average output power increases almost linearly. The maximum output power is measured to be 5.11 mW at the pump power of 600 mW.

## Conclusion

In conclusion, we have reported the  $\text{MoS}_2/\text{SiO}_2$  composite materials, which are prepared by incorporating the  $\text{MoS}_2$  nanomaterials in sol-gel glass. EDS spectrum identifies the main component of prepared  $\text{MoS}_2/\text{SiO}_2$  glass. The modulation depth and saturable intensity of  $\text{MoS}_2/\text{SiO}_2$  composite materials are measured to be 3.5% and  $20.15 \text{ MW}/\text{cm}^2$ , respectively. Mode-locked fiber laser with  $\text{MoS}_2/\text{SiO}_2$  is further demonstrated. The conventional soliton mode-locking state with a pulse duration of 780 fs is realized at the pump power of 90 mW. In the pump power range of 100–600 mW, another stable mode-locking state is presented. The pulse width is 1.21 ps and the maximum output power is 5.11 mW. Our results show that the  $\text{MoS}_2/\text{SiO}_2$  composite materials possess a good prospect in ultrafast photonics and the sol-gel method provides a new way for fabrication of TMD optical devices.

## Abbreviations

2D: Two-dimensional; CVD: Chemical vapor deposition; EDF: Er-doped fiber; EDS: Energy dispersive X-ray spectrometer; FWHM: Full width at half maximum;  $I_{\text{sat}}$ : Saturable intensity; LD: Laser diode; LPE: Liquid phase exfoliation; ME: Mechanical exfoliation; MSD: Magnetron sputtering deposition; PC: Polarization controller; PH-SO: Polarization independent isolator; PLD: Pulsed laser deposition; SA: Saturable absorber; SESAM: Semiconductor saturable absorber mirror; SNR: Signal-to-noise ratio; TEOS: Tetraethoxysilane; TMD: Transition metal dichalcogenide; WDM: Wavelength division multiplexer;  $\Delta T$ : Modulation depth

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## Availability of Data and Materials

Not applicable.

## Authors' Contributions

LL performed the laser experiments, analyzed the data, and wrote the paper. RL performed the nonlinear optical properties experiments. ZC prepared the  $\text{MoS}_2/\text{SiO}_2$  composite materials and interpreted the data for SEM image and EDS spectrum. JW and SL contributed to the scientific discussion. WR and YW conceived and designed the experiments. All authors read and approved the final manuscript.

## Competing Interests

The authors declare that they have no competing interests.

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